A RATIONALE FOR DEFORMATION TWINNING IN NANOCRYSTALLINE MAGNESIUM AND MAGNESIUM AZ80 ALLOY

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Abstract

Recent reports have shown that the generation of nanotwins in nanostructured grains can further enhance both the strength and ductility of fcc alloys; in such alloys, twinning can be far more prominent in nanoscale grains than for their coarse-grained counterparts where deformation twinning rarely occurs. Mg presents a more perplexing challenge, as the opposite phenomena occurs; twinning is a prevalent deformation mode for coarse-grained materials, but has been observed to occur more infrequently as the grain size decreases, and rarely at the nanoscale, which is reported to be due to the high stress needed to nucleate a partial dislocation from a boundary, and the lower energy needed for dislocation slip to preferentially occur. In this preliminary report, we demonstrate that the unique thermomechanical processing conditions offered by cryomilling result in the nucleation and growth of deformation twins in nanostructured grains. It is hypothesized that the high rate of deformation, combined with the reduced temperature facilitate the high stresses needed for twins to nucleate at a grain boundary. Transmission electron microscopy and the corresponding nano-diffraction patterns show evidence of the same compression twinning systems that occur in coarse-grained materials. These results point to a promising approach for the design of nanocrystalline Mg-alloys with superior strength and ductility for advanced structural applications.

Introduction

Nanocrystalline metals have shown mechanical properties significantly better than their coarse-grained counterparts [1]. Recent reports by Lu et al. have shown large increases in both strength and ductility through the introduction of dense, thin nanotwins into nanocrystalline Cu grains [2,3]. While the mechanism for twinning in nanocrystalline fcc materials is well-studied and understood [4], the mechanisms for twinning in nanocrystalline hcp materials remain heretofore unclear [5]. Most importantly, twinning is known to be difficult in coarse-grained fcc alloys, and shown to be more prevalent as the grain size decreases [4]. Conversely, given the limited number of slip systems active in room temperature hcp alloys, twinning plays an important role in plastic deformation of coarse-grained materials [6,7]. However, twinning has been observed to occur less as the grain size decreases, and observed infrequently in sub-micrometer grains [8-10].

It has been reported that twins are not observed in fine-grained Mg alloys due to the high local stress needed to nucleate a partial dislocation at a grain boundary [8,11,12]. However, recent reports have observed twinning in nanocrystalline Mg-alloys processed by high-energy milling [13-16]. In [15], it is postulated that the addition of 10 at.%Ti reduced the stacking fault energy of the system, thus facilitating easier twinning during the ambient temperature milling. The work reported in [16] shows preliminary evidence of twinning in cryomilled AZ80 alloy, but does not

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14. ABSTRACT

Recent reports have shown that the generation of nanotwins in nanostructured grains can further enhance both the strength and ductility of fcc alloys; in such alloys, twinning can be far more prominent in nanoscale grains than for their coarse-grained counterparts where deformation twinning rarely occurs. Mg presents a more perplexing challenge, as the opposite phenomena occurs; twinning is a prevalent deformation mode for coarse-grained materials, but has been observed to occur more infrequently as the grain size decreases, and rarely at the nanoscale, which is reported to be due to the high stress needed to nucleate a partial dislocation from a boundary, and the lower energy needed for dislocation slip to preferentially occur. In this preliminary report, we demonstrate that the unique thermomechanical processing conditions offered by cryomilling result in the nucleation and growth of deformation twins in nanostructured grains. It is hypothesized that the high rate of deformation, combined with the reduced temperature facilitate the high stresses needed for twins to nucleate at a grain boundary. Transmission electron microscopy and the corresponding nano-diffraction patterns show evidence of the same compression twinning systems that occur in coarse-grained materials. These results point to a promising approach for the design of nanocrystalline Mg-alloys with superior strength and ductility for advanced structural applications.

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discuss the nature of the twins generated, and largely focuses on the mechanical response of the consolidated nanocrystalline AZ80 powder. In this report, we utilize a novel transmission electron microscopy (TEM) analysis approach to interrogate the powders generated in [16] in addition to commercially pure Mg (99.9%) processed under similar conditions to elucidate the nature of twin formation in nanocrystalline Mg and Mg-alloys. We postulate that the low temperature and high-strain rates during cryomilling offer an environment where a partial dislocation can be nucleated from the boundary of a nanocrystalline grain. Unsurprisingly, the same {10-11} compression twins that are observed to assist in the plasticity of coarse-grained Mg alloys are evident in nanocrystalline Mg and Mg-alloys.

Experimental Methodology

For this study two alloys were selected: commercially pure (99.9%) pure Mg powder with an average spherical particle size of 75 μm, and a AZ80 Mg-alloy (7.82 wt.%Al, 0.47 wt.%Zn and 0.16 wt.%Mn) with an average particle size of 55μm. Both materials were cryomilled in liquid Ar at temperatures of -186°C in a modified Szegvari Attritor mill with 6.4 mm steel balls in a steel canister. The powders were milled for using an impellor speed of 180 rpm for 8h with a ball to powder ratio of 45:1 for the pure Mg, and 60:1 for the AZ80.

Transmission electron microscopy (TEM) was performed to determine the resultant grain size after milling, and selected area diffraction patterns were obtained to elucidate the nature of the deformation twin structures generated during the cryomilling. TEM samples of cryomilled pure Mg were prepared by directly spreading the powder onto a Cu grid with carbon film. The Cu grid was dipped into ethanol and dried before the spreading process. For the AZ80, precompacted cryomilled powder [16] was hand-ground and subsequently spread onto a Cu grid. TEM images and nano-beam diffraction patterns were obtained using a 200KV JEOL JEM-2000FX microscope.

With conventional TEM grain size analysis, efforts are made to focus on grains within the surrounding large (μm-scale) particle. In this study, we take advantage of the fact that grains fragment away from the edges of large particles when they are exposed to the electron beam (see Fig. 1). The primary benefit of this method of grain analysis is that grain overlap is much less likely to occur, and thus analysis of individual grains is facilitated, and the corresponding nano-diffraction patterns used to determine the deformation twin types might be clearly interpreted.

Results

It has been shown elsewhere [16] that the average particle size in the cryomilled AZ80 is reduced from 55 μm to ~18 after 8h of cryomilling at -186°C. Similarly, while not shown here for brevity, the particle size is reduced from 55 μm to ~20 μm in the cryomilled pure Mg. Given the limited slip systems, and thus the brittle nature of Mg and its alloys, especially at low temperatures [17], it is expected that some of this size reduction is a result of particle fracture. However, the size reduction is not large, and significant plastic flow is evident from the presence of nanocrystalline grains in the post-cryomilled particles. TEM images of these nanocrystalline grains are presented in Fig. 2.

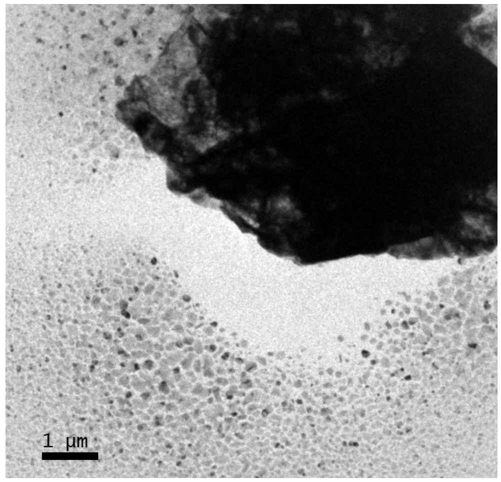

Figure 1: TEM image of pure Mg showing fragmented nanocrystalline grains surrounding the original microcrystalline cryomilled particle.

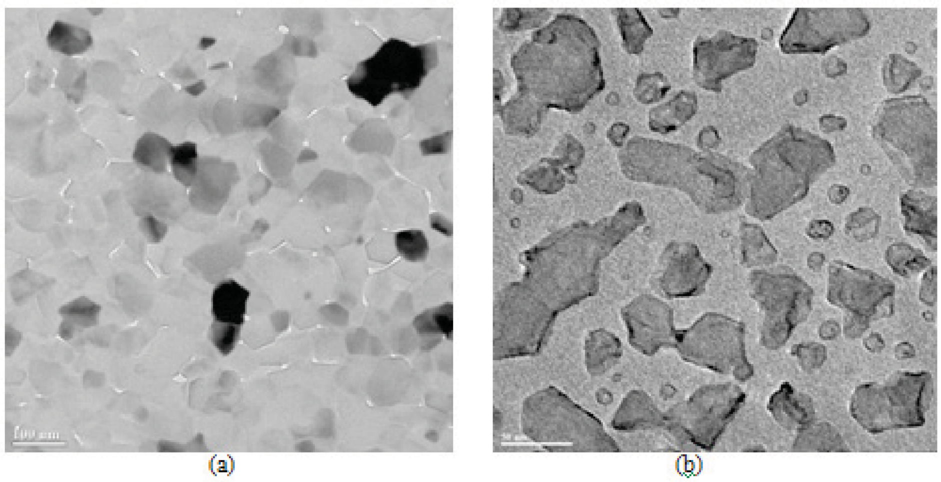

Figure 2: TEM images of cryomilled (a) pure Mg with an average refined grain size of 52 nm (100 nm scale bar), and (b) AZ80 with an average grain size of 40 nm (50 nm scale bar) after 8h of milling at -186°C.

Grain size analysis of the TEM images in Fig. 2, and a number of other similar images from the same samples showed the average grain size of the Mg and AZ80 to be 52 nm and 40 nm respectively after 8h of cryomilling. Nieh and Wadsworth [18] estimated the minimum grain size in which a metal could exist without supporting dislocations by equating the repulsive force between the applied stress and dislocations. Using this estimation, an analysis presented by Hwang et al. [19] shows the upper and lower bounds for the minimum grain size to be 30 and 60 nm, respectively, for Mg alloys, which is in agreement with the values we currently report.

Further TEM interrogation of the nanocrystalline Mg and AZ80 grains for twins was performed, and some corresponding images and nano-diffraction patterns are shown in Figs. 3 and 4. Both materials show evidence for {10-11} type compression twins that are typical in deformed coarse-grained Mg-alloys [7].

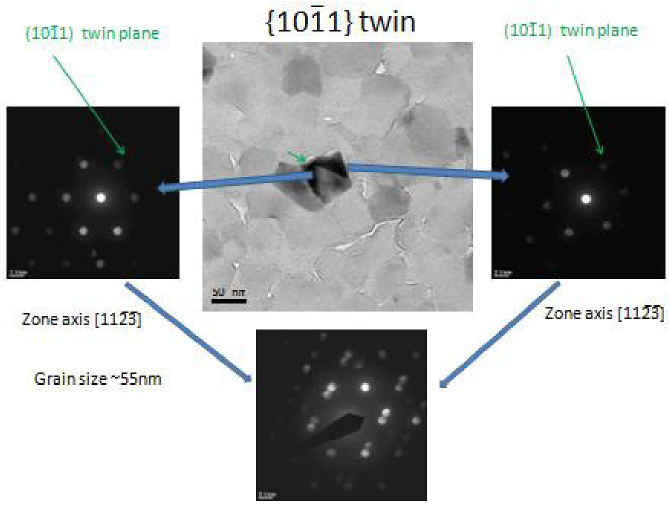

Figure 3: TEM image of a nanocrystalline (~55 nm) pure Mg grain and corresponding nano-diffraction patterns ([11-2-3] zone axis) showing evidence for a {10-11} type compression twin (gray arrow).

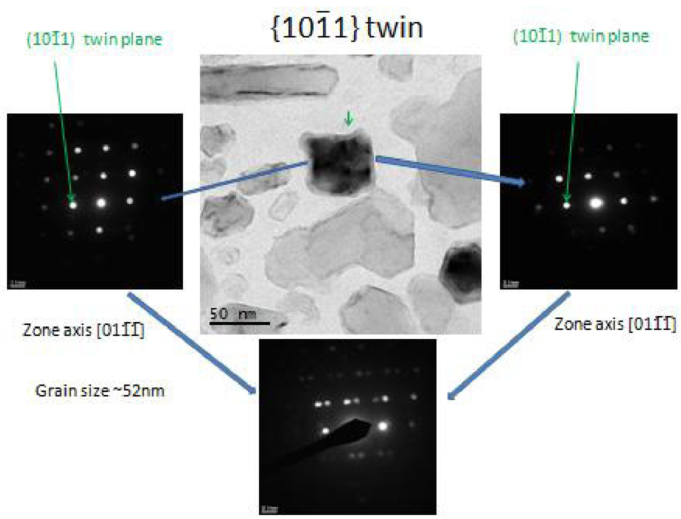

Figure 4: TEM image of a nanocrystalline (~52 nm) AZ80 grain and corresponding nano-diffraction patterns ([01-1-1] zone axis) showing evidence for a {10-11} type compression twin (gray arrow).

Discussion

In the Results section, we present clear evidence for the formation of {10-11} compression twins during the cryomilling of Mg and AZ80 alloy not unlike those observed to occur in coarse-grained Mg-alloys [7]. This analysis supports preliminary findings of such twins in [15], and are also predicted in recent molecular dynamic simulations [20]. These findings do not invalidate the prior reports that deformation twins are not observed in fine-grained Mg-alloys [8-10]. However, the lack of observation should not be misinterpreted as the inability for such materials to deform by deformation twinning. Accordingly, we postulate a rationale as to why such twinning occurs in nanocrystalline Mg processed by cryomilling.

While the fundamental physics of twin nucleation and growth are not clearly understood in the case of hcp materials, the fundamental physical mechanism for twin nucleation should be through generation of a partial dislocation from a grain boundary. It is widely understood that for hcp Mg-alloys the suppression of twinning is due to the high twin nucleation stress [10, 21, 22] that accompanies the fine-grains. But the thermomechanical conditions under which Mg-alloys are deformed [8-10, 23], i.e., quasi-static deformation rates ($< 10^{-2}$ /s) and $T > T_{ambient}$ should not realistically be expected to result in high local stress at a grain boundary. The thermomechanical conditions present during cryomilling, on the other hand, involve high-strain-rate deformation ($>10^3$ /s) at very low temperature (< -150°C).

In the seminal work by Christian and Mahajan on deformation twinning, it is reported that deformation at reduced temperatures and high-strain rate can independently facilitate the nucleation stresses needed for twinning via suppression of slip and other deformation mechanisms [5]. This mechanism for inducing deformation twinning was first reported experimentally in nanocrystalline Al-alloy processed by cryomilling [24]. Moreover, a very high dislocation density was observed around twinned boron carbide particles within nanostructured Al5083/B_4C metal matrix composite processed by cryomilling, which supports the assumption that a very high local stress exists, and could nucleate a twin in high-strength boron carbide particles [25]. Given these observations, it should be expected that under similar extreme thermomechanical processing conditions, that shear stresses on a grain boundary, combined with the suppression of other deformation mechanisms, would enable deformation twinning to occur as observed in Mg and Mg-alloys, and a recently reported probabilistic description of twin nucleation at Mg grain boundaries supports this interpretation as well [10].

While not discussed at length here, there is clearly a competition between the stress needed to deform the nanocrystalline Mg-alloys by slip and other deformation modes, and the stress needed to nucleate a deformation twin [4, 10]. It is not clear under which conditions the twin nucleation stress will be less than that needed for competitive slip process. Factors such as textural suppression of slip [26] and impurities in the grain boundaries that may hinder slip and grain boundary sliding [27] might play important roles that are functions of ultrafine (< 1μm) grain size, deformation temperature and deformation rate, and therefore this work will be targeted for future studies.

Conclusions

Commercially pure Mg and AZ80 Mg-alloy were processed by cryomilling to grain sizes in the nanocrystalline regime (<100 nm). Transmission electron microscopy and corresponding nano-diffraction patterns of both materials reveal compression-type {10-11} twins that are commonly observed in deformed coarse-grained Mg-alloys. It is postulated that the low-temperature and high-strain-rate thermomechanical processing conditions offered by cryomilling provide a unique environment wherein enough stress can be generated on a nanocrystalline grain boundary to nucleate a partial dislocation that subsequently grows into a twin. These observations offer insights into the deformation mechanisms of nanocrystalline Mg-alloys, and forecast the ability to design and synthesize Mg-alloys with improved mechanical properties.

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